Development of technology for formation of Al-B-N system joints under influence of high pressures and temperatures to create composite tool material

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Abstract. Investigation of formation processes and characteristics of Al-B-N system compounds obtained under the influence of high pressures (up to 8 GPa) and temperatures (up to 2000K) to create composite tool materials on their basis. The main scientific idea is that a method will be developed for forming compounds of the Al-B-N system under the action of high pressures and temperatures. The obtained compounds of this system will create the prerequisites for creating composite tool materials on their basis for specific application tasks in the industry.

1 Introduction

The current level of development of technology requires the use of new materials. To create components, mechanisms, and assemblies that operate for a long time at high speeds and heavy loads, it is necessary to use materials with improved characteristics (strength, hardness, wear resistance, etc.). The need for materials with improved characteristics for various applications worldwide grows rapidly yearly. The combination of high hardness and crack resistance is of fundamental importance for the efficient operation of the tool in highly loaded operating conditions. Improving the mechanical and performance properties of the tool is a priority for efficient tool operation over a wide temperature range. In this regard, the use of cubic boron nitride is topical.

Aluminum nitride strongly influences synthesizing cubic boron nitride as a catalyst. Such a nitride was obtained in [1-5], which used a mixture of BN:Al 9:1 paint, sintered at high pressure, and annealed at 950 ^{0} C, pressure 0.003 Pa for 1 hour. As a result, a layer of columnar AlN grains 0.04 μm thick was obtained, and polycrystalline phases AlB10 and AlB12 were found at the interface.

In one of the first works [2, 6-11] on the study of the action of AlN when aluminum nitride is added to BNg in an amount of 20 mol. % and the synthesis of samples at a pressure of 6.5 GPa and a temperature of 1600 C in an inert or reducing atmosphere,

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according to the authors, almost all hexagonal boron nitride was transformed into a cubic one and had the shape of a typical tetrahedron with a grain size of 2 μm. The mechanism of BNc synthesis was also studied [3, 12, 13, 14]. In the case of the reaction in the presence of aluminum, the BNh - BNc transition occurs in the absence of a liquid phase, and hexagonal boron nitride dissolves in solid aluminum nitride and supersaturates it, while cubic boron nitride precipitates from such a supersaturated solution. The activation energy of such a transformation was 170 ± 40 kJ/mol. In work [4, 15, 16], the authors concluded that the limitation of the diffusion of boron and nitrogen atoms in aluminum nitride is the reason for the completion of the BNc formation reaction. They also give a hypothetical AlN-BN phase diagram at 6 GPa.

2 Methods

Investigation of methods for obtaining new composite materials based on compounds of the Al-B-N system using high pressure (up to 8 GPa) and temperatures (up to 2000 K), investigation of their physical characteristics for creating instrumental materials on their basis.

When exposed to high pressures of 5.5 GPa and 1750-1800 ºC temperatures, monocrystalline powder superhard materials are formed with specified characteristics depending on their further use (cutting, grinding, or polishing tools).

The high-pressure device of the "anvil with a hole" type (Figure 1) consists of two counter-moving hard-alloy dies 1 (made of VK-6), fastened along the side hazards with steel thrust rings 2 (steel 35 ХГСА). The flat end surfaces of the matrix are in contact with support plates 3 (hard alloy VK-15), which are supported by retaining rings 4. A container made of lithographic stone or pyrophyllite with a graphite heater and a solid mixture tablet is placed inside a matrix. When loaded into a container, a quasi-hydrostatic state is measured in units of hydrostatic pressure. The reaction mixture is heated by passing an electric current through a graphite heater. The high density of the heat flux passing through the carbide dies, which are in thermal and electrical contact with the heating element, leads to their rapid heating. Therefore, to control the temperature, the apparatus was placed in a water-cooled case (the arrows indicate the direction of the water inlet and outlet). This ensures the stability of the thermal regime in the reaction volume. The rotameters of the RS-5A type regulate the water pressure. To ensure a stable temperature regime, the press unit DO 137 A was additionally equipped with an OPTRON synthesis programmer, which allows you to regulate and stabilize the temperature with an accuracy of +10°K.

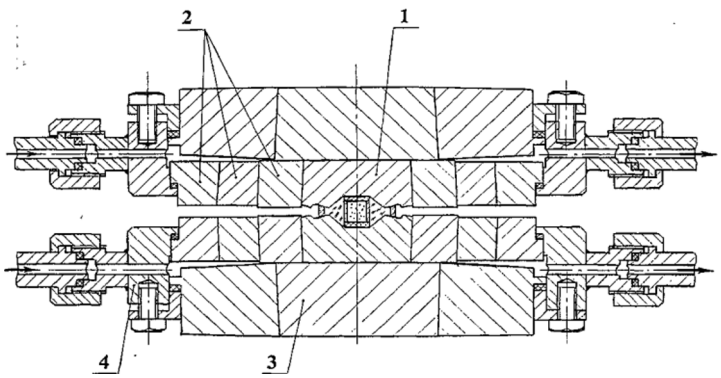

Fig. 1. High-pressure device for the synthesis of superhard materials: 1 is carbide dies with a container and a heating element; 2 is steel supporting rings; 3 is carbide base plates with supporting rings; 4 is housing for water cooling (arrows indicate the direction of water inlet and outlet).

Pressure regulation in the reaction volume of the high-pressure apparatus. SVD calibration by pressure in coordinates: pressure in the reaction volume (GPa) – pressing force (kgf/cm^2) (Figur 2). The AED calibration curves used in this work were built by recording the change in the electrical resistance of the reference material during its polymorphic transformation with a change in pressure in the chamber. Calibration was carried out at room temperature by phase transitions of solid solutions of sulfides and selenides of zinc and cadmium CdS 2.4 GPa, $Zn_{0.05}Cd_{0.95}S$ 3.5 GPa, $Zn_{0.23}Cd_{0.77}S$ 5.3 GPa, CdSe 2.5 GPa, $Zn_{0.07}Cd_{0.93}Se$ 3.3 GPa, $Zn_{0.13}Cd_{0.87}Se$ 4.1 GPa, $Zn_{0.2}Cd_{0.8}Se$ 4.6 GPa, $Zn_{0.3}Cd_{0.7}Se$ 5.6 GPa.

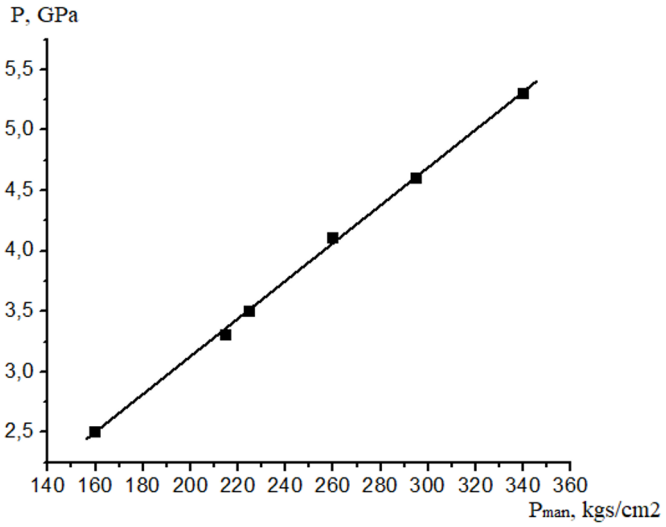

Fig. 2. Dependence of pressure created in HPA on force of press DO 137A

The reaction mixture for synthesis, consisting of powders, was stirred for 20 h in a mixer with an offset axis. Then, under a pressure of 0.25 GPa, cylindrical tablets with a diameter of 10 and a height of 2 mm were pressed from the mixture in a steel mold at room temperature and placed in a composite heater pressed from a charge consisting of graphite and BNh, located in a high-pressure chamber, after what the synthesis was carried out.

Fig. 3. High-pressure apparatus DO 137A.

The synthesis was carried out at a pressure of 5 GPa (taking into account the thermal pressure increase equal to ~ 0.8 GPa) and temperatures of 1670-1910 K in high-pressure carbide chambers of the "anvil with a hole" type in containers made of lithographic stone. With the given synthesis parameters, 5 identical experiments were carried out.

The installation for the synthesis of compounds in the B-N-Al and B-N-Al-Ti system at high pressure and temperature includes a hydraulic press DO 137 A, a high-pressure apparatus (HPA), and a power transformer for electrical heating of the reaction volume, high-pressure chambers and OPTRON synthesis programmer. Synthesis was carried out in TPS with hard-alloy matrices of the "anvil with a hole" type.

At room temperature, phase formation studies were conducted on DRON-3 and DRON-3-M diffractometers in Cr-Kα and Cu-Kα radiation. A graphite monochromator was used to cut off the Kβ component of the radiation. The scanning step is not more than 0.03 degrees; the exposure time is not less than 5 seconds. The data was captured automatically. The full-profile analysis of X-ray diffraction data studied the crystal structure according to the Rietveld method. The spectra were refined using the FullProf software package.

Obtaining samples of the B-N-Al system was carried out from elemental Al and hexagonal modification BN (in the ratio Al:BN=0.75:0.25 by weight) under high pressure equal to 5.0 GPa, at various temperatures from 500˚C to 2000. C at a synthesis duration of 3 min in high-pressure carbide chambers of the "anvil with a hole" type in containers made of lithographic stone. The duration of the synthesis is set experimentally. The chosen synthesis conditions make it possible to obtain compounds of this system with a high degree of phase purity.

The data of X-ray diffraction analysis of the obtained samples of the B-N-Al system indicate that in the sample, starting from a temperature of 1500˚C (inclusive) and up to 2000˚C, the AlxNy phase is formed and there is a phase of transition of hexagonal BN to the cubic modification (Figs. 4 and 5). Along with the AlxNy phase, the samples contain other compounds of the B-N-Al system due to the initial charge's non-stoichiometric ratio.

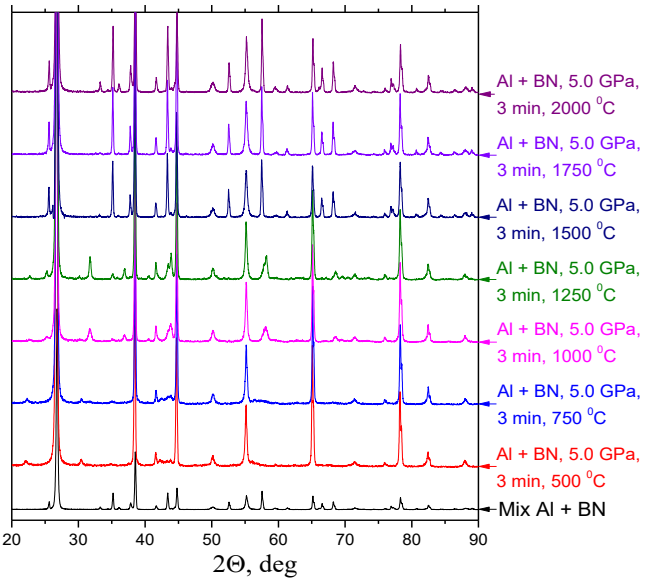

Fig. 3. X-ray diffraction spectra of obtained samples of B-N-Al system obtained under high pressure of 5.0 GPa at various temperatures of 500 - 2000°C with a synthesis duration of 3 minutes

Obtaining samples of the B-N-Al-Ti system was carried out from elemental Al, Ti, and hexagonal modification BN (in the ratio Al:Ti:BN=0.25:0.25:0.50 by weight) at various high pressures from 2.0 to 5.0 GPa, at various temperatures from 500˚C to 2000˚C with a synthesis duration of 3 min in high-pressure carbide chambers of the "anvil with a hole" type in containers made of lithographic stone. The chosen synthesis conditions make it possible to obtain compounds of this system with a high degree of phase purity.

The data of X-ray diffraction analysis of the obtained samples of the B-N-Al system indicate that in the sample, starting from 2.0 GPa and up to 5.0 GPa (Fig. 4) at a temperature of 1500˚C and 2000˚C, AlxNy, the TixNy phase is formed, and the phase transition of the hexagonal modification of BN to the cubic modification of cBN occurs. Along with the AlxNy and TixNy phases, the samples also contain other binary compounds of the system under study.

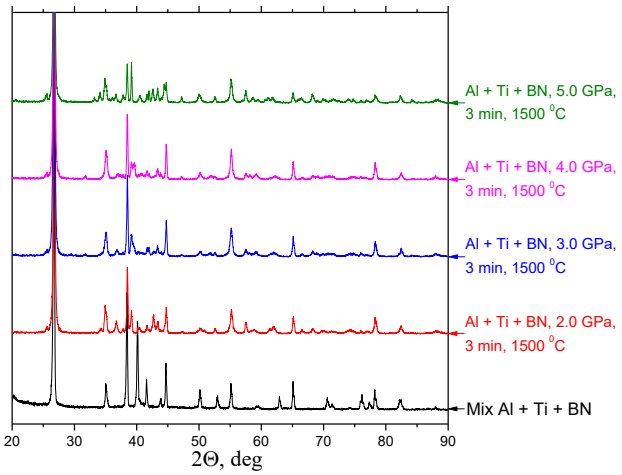

Fig. 4. X-ray diffraction spectra of obtained samples of B-N-Al-Ti system obtained at various high pressures from 2.0 GPa to 5.0 GPa at 1500°C with synthesis time of 3 minutes

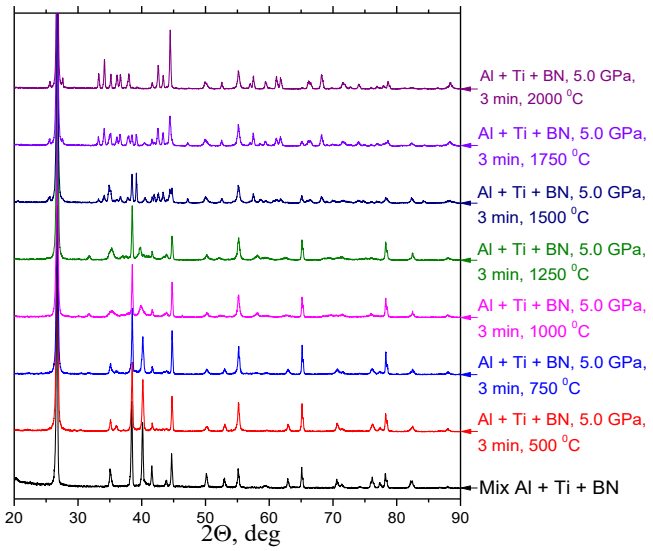

Fig. 5. X-ray diffraction spectra of obtained samples of B-N-Al-Ti system obtained under high pressure of 5.0 GPa at various temperatures of 500 - 2000°C with synthesis duration of 3 minutes

Obtaining samples of the B-N-Al system was carried out from elemental Al and hexagonal modification BN (in the ratio Al:NB=0.50:0.50 by weight) at various high pressures from 1.0 to 5.0 GPa, at various temperatures from 500 ˚C up to 2500°C with a duration of synthesis of 3-4 min in hard-alloy high-pressure chambers of the "anvil with a hole" type in containers made of lithographic stone. The chosen synthesis conditions make it possible to obtain compounds of this system with a high degree of phase purity.

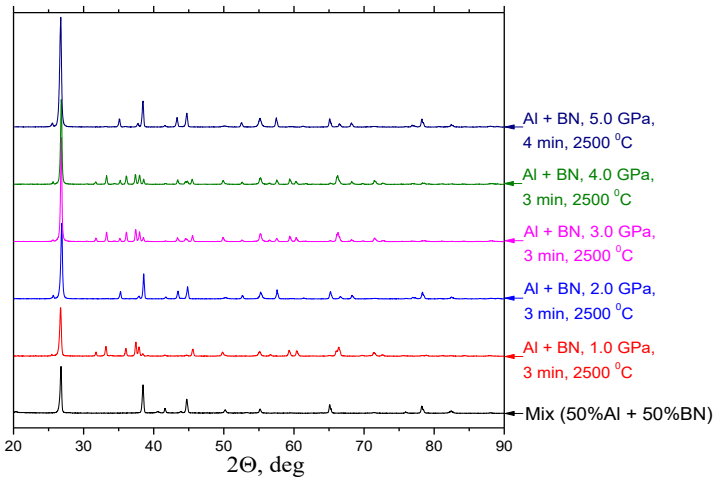

Fig. 6. X-ray diffraction spectra of the obtained samples of the B-N-Al system obtained at various high pressures from 1.0 GPa to 5.0 GPa at a temperature of 2500˚C with a synthesis duration of 3-4 minutes

The data of X-ray diffraction analysis (Figure 5) of the obtained samples of the B-N-Al system indicate that in the sample, starting from 1.0 GPa and up to 5.0 GPa at a temperature of 2500 ° C, the AlxNy phase is observed, AlxBy has formed and a phase transition of the hexagonal NB. in the cubic modification of CNB. Along with the AlxNy and AlxBy phases, the samples also contain other binary compounds of the system under study.

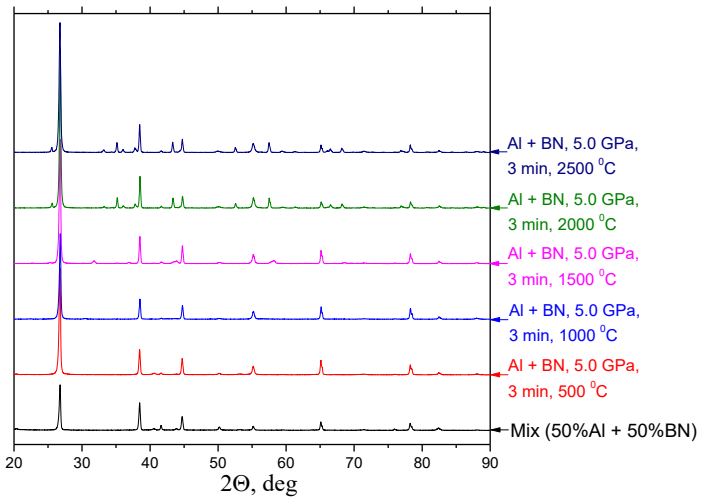

Fig. 7. X-ray diffraction spectra of the obtained samples of the B-N-Al system, obtained under high pressure of 5.0 GPa at various temperatures of 500 - 2500 ° C with a synthesis duration of 3 minutes

The data of X-ray diffraction analysis (Figure 7) of the obtained samples of the B-N-Al system indicate that in the sample, starting from 1500˚C and up to 2500˚C (inclusive), at a high-pressure value of 5.0 GPa and with a synthesis duration of 3 minutes, the formation of AlxNy, AlxBy phases and the phase transition of hexagonal NB to the cubic modification cNB occurs [17]. Along with the AlxNy and AlxBy phases, the samples also contain other binary compounds of the system under study, which is associated with the nonstoichiometric ratio of the initial charge.

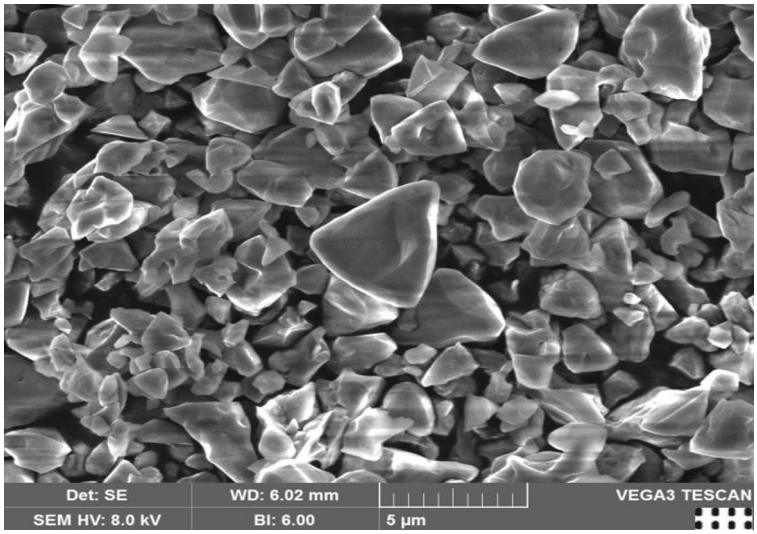

Fig. 8. Microstructure of cBN samples, sintered at pressure of 8 GPa

Table 1. Mechanical properties of composite materials based on cubic boron nitride

№	Name	Value	
1	Hardness	30-45	GPa
2	Crack resistance	12-16	MPa•$m^{1/2}$
3	Resistance when turning hardened steels (HRC 52-54)	60	min.
4	Grain size	5-30	mcm
5	Depth of cut up to	0.5	mm
6	Feed	0.05 – 0.15	mm/tur.
7	Cutting speed	200 – 125	m/min.

Experimental studies of the cutting properties of cutters based on B-N-Al system joints were carried out at the Tashkent Diesel Locomotive Repair Plant and the Tashkent Foundry and Mechanical Plant. The studies were carried out by changing the cutting tool operating mode parameters during the finishing of parts.

Fig. 9. Obtained high-pressure synthesis samples (without machining)

At the "Tashkent Diesel Locomotive Repair Plant", the shaft Ø 57 mm of a blower made of steel grade 12XN3A with a hardness of 56 HRC, with the parameters of the cutter operating mode: cutting feed 0.05 mm/rev, cutting depth 0.1 mm, cutting speed 285 m / min. The working time of the cutter was 88 minutes (Figure 9).

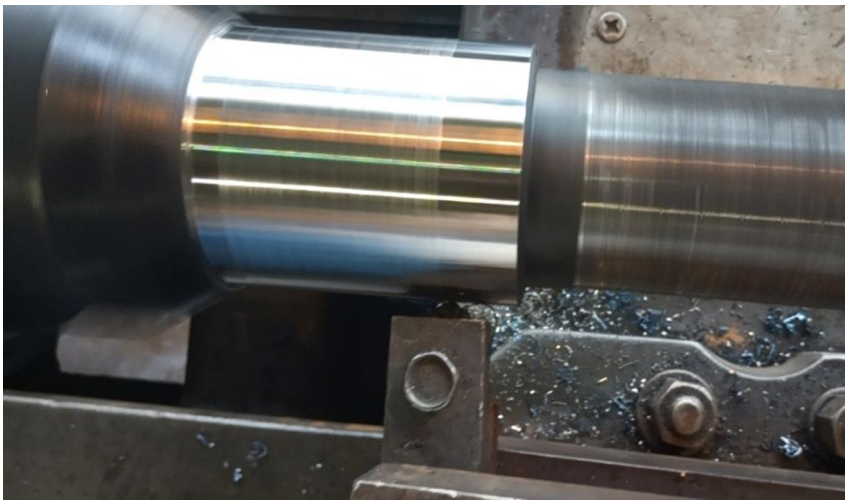

Fig. 10. Turning shaft Ø 57 mm from steel grade 12XN3A with cutter feed of 0.05 mm/rev and cutting speed of 285 m/min

External longitudinal turning of a shaft Ø 57 mm was carried out with the following mode parameters: tool feed 0.15 mm/rev, cutting depth 0.1 mm, cutting speed 229.1 m/min. The working time of the cutter was 82 minutes (Figure 11).

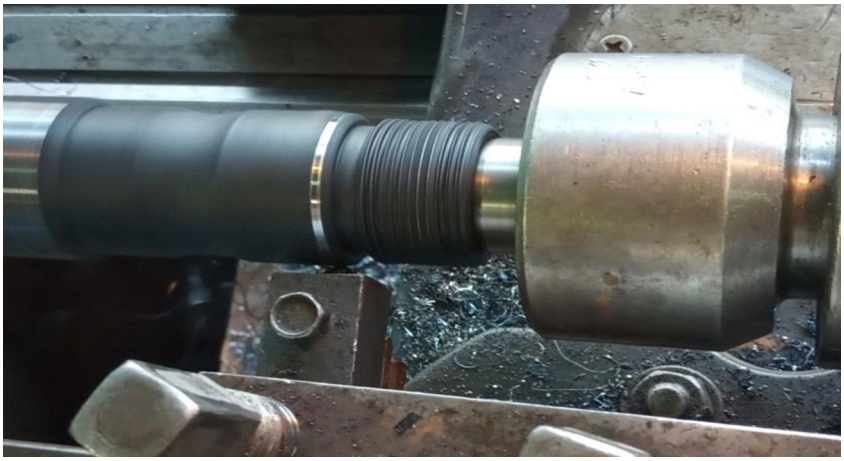

Fig. 11. External longitudinal turning of shaft Ø 57mm from steel grade 12XN3A cutter feed 0.15 mm/rev and cutting speed 229.1 m/min

3 Conclusions

The obtained composite compounds are a prerequisite for creating a binder based on the B-N-Al system for specific industry use tasks.

Based on several system connections, changing the composition and not significantly adjusting the technological process, it is possible to quickly obtain instrumental composite materials of various directions.

Several mechanical tests have been carried out on various grades of steel. At the same time, the working time of the cutters (durability) was 78 - 88 minutes.

Experimental studies of cutting properties of cutters based on B-N-Al system joints have been carried out. Recommendations on rational conditions and modes of cutting tools using superhard composite tool materials have been developed.

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